

Reagents Modification Study for Improved Flotation of a Gold Ore at the Dundee Precious Metals Krumovgrad Mine, Bulgaria

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ABSTRACT: A laboratory study was conducted with the view of replacing the currently used secondary collector (promoter) at the Dundee Precious Metals, Krumovgrad (Bulgaria) flotation plant with a more efficient one securing better recovery of the electrum from the ore. Eight reagents from various suppliers have been tested to benchmark their performance against the currently used promoter. The promoters were compared based on concentrate mass pull and gold and silver grade and recovery. Three of them have shown superior performance on a comparative basis and have been subjected to further optimization. SEM inspection of the precious metals grains lost in tailings was completed in parallel. The effect of reagents dosage on precious metals grades/recovery levels achieved by the best performing promoters is discussed.

Keywords: Gold ore, flotation reagents, quantitative mineralogy, grade-recovery

INTRODUCTION

Study Context

At Krumovgrad, Bulgaria, the mining operation managed by Dundee Precious Metals of Canada extracts and processes an ore from the Ada Tepe deposit which is classified as a low sulphidation epithermal gold deposit (Marchev et al., 2003, 2004; Tsintsov & Ivanov, 2012). The majority of gold present in the deposit is in the form of electrum. The Krumovgrad plant processes the ore through crushing, grinding and different flotation stages to ultimately yield a precious metals concentrate. Being a classical flotation operation, plant metallurgical performance depends

amongst others, largely on the physical characteristics of the precious metals mineral carriers, like particle size, shape and degree of liberation (Allan & Woodcock, 2001). Therefore an automated mineralogy investigation in conjunction with batch flotation testing was envisaged as an enabling tool to understand the intrinsic behavior of gold in flotation.

On the other hand, the type of reagents used and their performance can be influenced by the physicochemical parameters of the pulp. Xanthate group collectors are normally the preferred choice in gold flotation with their performance being largely dependent on the redox potential

governing its adsorption onto the mineral surface. A certain amount of oxygen in the pulp is required to allow the formation of di-xanthogen and addition of secondary promoters such as dithiophosphates to provide a synergetic action with xanthates is also practiced (Chanturia et al., 2012, Dunne, 2005). Often the impact from the synergetic effect is an increase in recovery, however sometimes at the expense of grade.

Since the principal precious metals carrier in the ore is electrum, it is necessary to likewise focus on the role of the gold/silver ratio when discussing electrum floatability. Therefore special attention in this study was devoted to the Ag grade in the precious metal grains which report to the concentrate and tailings under the action of the reagents tested.

Deposit Geology

The Ada Tepe deposit is situated in the East Rhodope region, a section of the Rhodope metallogenic province, which hosts a series of volcanic epithermal and base metal vein deposits. Ada Tepe is characterized by a mineralization of low-sulfidation epithermal gold mainly composed of layers of breccia, conglomerates, sandstone, and limestone (Márton et al., 2010). The sediments are conformed above a low angle detachment fault also referred as the Tokachka fault, followed by a metamorphic unit (Marchev et al., 2004). The sediments are the principal source of gold in the deposit since they have undergone high tectonic deformation, resulting in high silicification and enrichment in gold and silver.

The ore mineralogy consists of electrum and subordinate pyrite with some traces of galena, and gold-silver tellurides (Hessite and Petzite). The gangue minerals comprise silica polymorphs like microcrystalline, sugary quartz, opaline silica, etc., carbonates like calcite, dolomite of siderite and adularia, compared to adjacent deposits witnessing high Au/Ag ratio and low base metals content (Marchev et al., 2003, Marchev et al., 2004, Marinova, 2008, Martón, 2010).

Gold Mineralization

Gold mineralization occurs in two different structures, the lower zone or 'Wall' and the upper zone (Tsintsov and Ivanov, 2016). The "Wall" is a highly silicified zone with a massive tabular body located above the detachment fault. Silicification in this area began with the deposition of massive white to light grey silica destroying the original rock except for some gneiss fragments. Average gold content is 7.3 g/t (Marinova, 2008).

The second type of mineralization in the Upper zone are open space-filling ores which are deposited within east-west oriented listric faults. They go through the whole sediments from their detachment fault to the surface. Their thickness varies between 0.10 to 0.80 m and can reach gold grades of 638 g/t (Marchev et al., 2003 and Marchev et al., 2004). Figure 1 presents samples corresponding to high-angle faults where colloform textures of quartz and adularia alternate with electrum layers bringing increased gold content.

Electrum Morphology and Size

Within the Wall zone the electrum is deposited normally in interstices of massive microcrystalline quartz, the morphology depending on the inherited geometry of these interstices. This situation can bring electrum to morph into complex dendritic forms when associated with quartz. However, in open spaces when liberated it can show a more globular shape (Marinova, 2008). The particles found are moderately flattened and almost isometrical and spherical (Marinova, 2007).

Electrum from the high-angle veins is deposited in colloform bands consisting of microcrystalline quartz, adularia, pyrite, and in some circumstances chalcedony. The thickness of the bands varies from tens of millimeters to 1 cm, rarely being thicker. This vein gold grade normally can span from 0.5 kg/t up to 7.899 kg/t commonly referred to as Bonanza gold. Also in these veins, electrum can occupy 50% of the volume (Marchev et al., 2004, Marinova 2008).

The size of electrum varies from 8 to 50 μm at the surface and from 1–4 to 25 μm in depth, 90% of the particles falling into that range, although in some cases particles between 100 up to 650 μm can also be observed (Marinova, 2007).

Additional studies have been done on placers from Ada Tepe, revealing that 95% of the grains were concentrated in fractions smaller than 100 μm . Some work on placer gold indicates that the higher gold content (45.32%) is found below 63 μm , and that the coarsest (100–125 μm) fraction reaches merely a concentration of 5.24% (Tsintsov and Popov, 2012).

Electrum Au/Ag ratio

Previous work reported that the electrum from the Ada Tepe has a ratio of Au/Ag of 3/1 (73–76% Au) for samples with gold content above 1% (Marchev, 2004). From a flotation recovery perspective one would assume that the reagents have to be adapted to deal with the various Au/Ag ratios of the electrum in the ore. This assumption is based on the fact that gold-silver alloys react with xanthate at different potentials, with ratios 80/20 Au/Ag reacting at

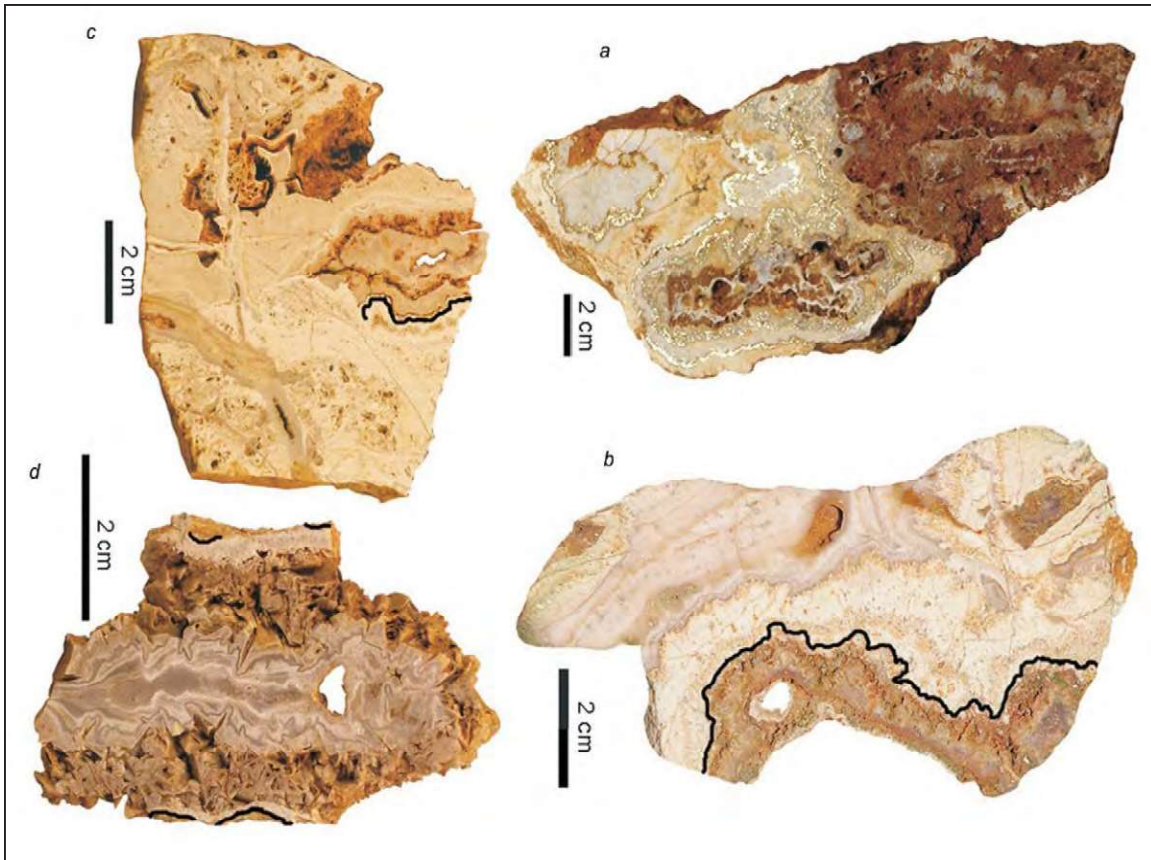


Figure 1. Colloform-banded macro-texture in hand specimens from high-angle veins Ada Tepe deposit (Marinova, 2013)

around 150 mV-SHE or -47 mV-Ag/AgCl. These potential figures gradually decrease with increasing silver grade until a very low potential value (-30 mV -SHE, -227 mV-Ag/AgCl) is reached for pure silver (Leppinen, 1991). In practice however, varying the type of the reagents employed is not realistic, therefore optimal blending of the ores to keep the mineralogy of the feed relatively constant is the preferred choice.

WORK OBJECTIVES

The objective of the work was to evaluate the response of the electrum in the ore against a series of commercially available flotation reagents, with an ultimate aim to replace the current Aerofroth®238 used as promoter at the plant. As a secondary aim, fine tuning of the dosage rates for the best performing reagents and investigating the reasons for precious metals losses still accountable in tailings was undertaken.

A previous study on Krumovgrad ore (Demeusy, 2023) showed a correlation between electrum recovery and Au/Ag ratio, notably by detecting a higher ratio of gold in the electrum grains in the concentrate, implying a negative effect

from the increased presence of silver. In light of these findings, the current work scope is to confirm the role of the Au/Ag ratio and its implication for metallurgical results.

As a rule, optimal floatability of gold is achieved when the redox potential is close to that of di-xanthogen formation. However for the case of the Ag-rich electrum in Krumovgrad tailings, the formation of Ag-xanthate complex occurs at lower potential (-20 mV) than the one between xanthogen and pure gold. Hence, evaluating the flotation performance of Au-rich- and Ag-rich-electrum at various pulp potential presents another goal of the work.

MATERIALS AND METHODS

Ore

The sample used in this study was a crushed run-of-mine ore sourced at the Krumovgrad mine with a P_{80} of 125 mm. It presents a blend of ore collected from the Wall zone and the Upper zone. In total 40 flotation tests have been completed (all in duplicate). The initially supplied batch of ore was sufficient to complete 24 tests, while for the remaining 16 tests a second batch of ore was supplied however with lower precious metals content. Table 1 gives the mean Au,

Ag and S content in the two batches back-calculated and averaged from the performed flotation tests. It can be seen that the batch one material is characterized by almost double the gold grade compared to the second batch. However, this difference did not significantly affect the overall results, with less than 2% recovery variation calculated.

Table 1. Mean Au, Ag and S grades with relative standard deviation, for the first (80 kg) and second (20 kg) batch of ore

	Element	Au, ppm	Ag, ppm	S, %
Batch 1	Mean	7.23	5.82	0.43
	RSD, %	3.68	5.9	9.5
Batch 2	Mean	4.01	4.03	0.33
	RSD, %	2.27	7.4	11.7

Communion

The ore was ground immediately prior to each flotation test using an instrumented Magotteaux® laboratory mill for 46 minutes to reach a P_{80} of 38 μm .

Flotation

The flotation machine used is a bottom-driven laboratory Magotteaux float Cell® equipped with a 5-L cell and fitted with temperature, pH, and redox potential probes. Pulp solids loading during flotation was about 30% with pH maintained at 8.3. Agitation was kept constant at 900 rpm for all tests. A flotation scheme was designed to simulate the rougher stage of the plant, replicating the exact dosage levels and reagents addition points. The main collector used was Potassium Amyl Xanthate (PAX), CuSO_4 was added as activator, sodium silicate—as dispersant and Aero-238®—as promoter. Figure 3 depicts the flotation sequence

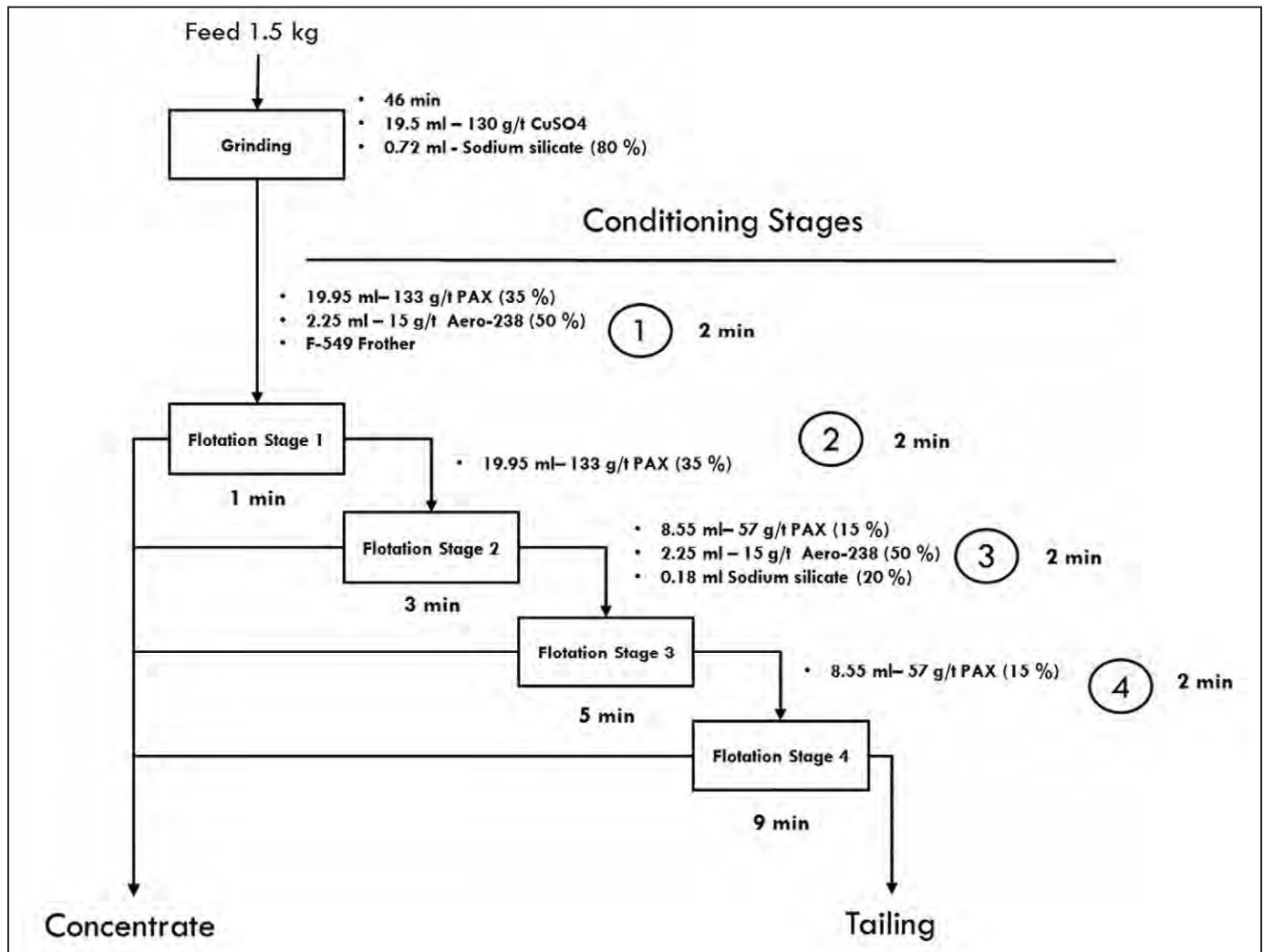


Figure 2. Flotation scheme chosen for testing, based on 1.5 kg of ore. Reference test involves Aero®238

showing details on the four conditioning and flotation stages lasting 18 minutes totally.

Characterization

Feed ore, concentrate and tailings have been assayed for Au, Ag and S through fire assay by the SGS branch located at DPM operation, Chelopech, Bulgaria.

Electrum occurrence was characterized in terms of mineral chemistry, morphology, size and degree of liberation. A SEM-based automated mineralogy system—Zeiss sigma 300 FEG with two Bruker EDS spectrometers was used to this end. Figure 2 illustrates typical electrum grains identified.

All identified grains in the feed ore were electrum, with no pure gold detected. Grain sizes varied from 2 to 20 μm ,

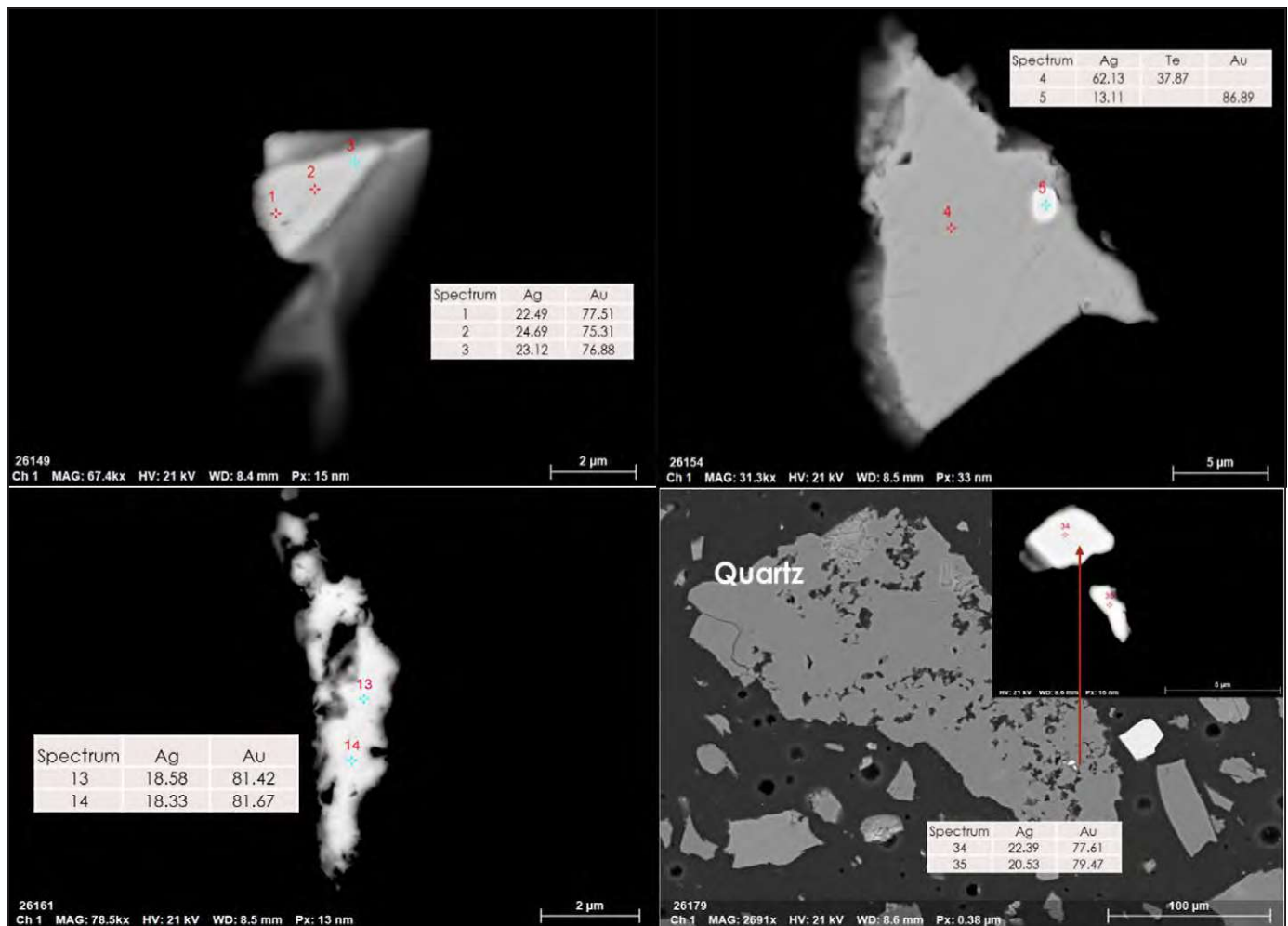


Figure 3. SEM-EDS images of electrum grains (composition and morphology) in the feed

Table 2. Flotation reagents (promoters) tested

Trade Name	Supplier	Chemistry (According to Data Sheet)
Danafloat-468	FMC Denmark	Dialkyl dithiophosphate
Danafloat-571	FMC Denmark	Dialkyl dithiophosphate and mercaptobenzothiazole
DP-OMC-1127	BASF	Anionic polyamine
Tecflote S11	Nouryon	Nitrile base collector
Armoflote S100	Nouryon	Amine Dithiocarbamate Sodium Salt
Hostafлот-10093	Clariant	Sodium dithiophosphate acid and phosphoric acid ester
Hostafлот-LIB	Clariant	Dithiophosphate acid and sodium salt
Hostafлот-7650	Clariant	Mixtures of thio-compounds

and they were mainly associated with quartz, orthoclase, iron oxides, and pyrrhotite. In a few particles, silver (50–60%) associated with tellurium was also detected.

Flotation

Experimental Design

The flotation work was divided into two phases. The initial screening tests aimed to select the best performing reagent. During the second phase, the best performing promoters were subjected to further optimization of dosage levels.

The initial screening encompassed eight secondary collectors/promoters listed in Table 2. The performance of each of them was benchmarked against the base case scenario (PAX - Aero-238® being the current practice on site).

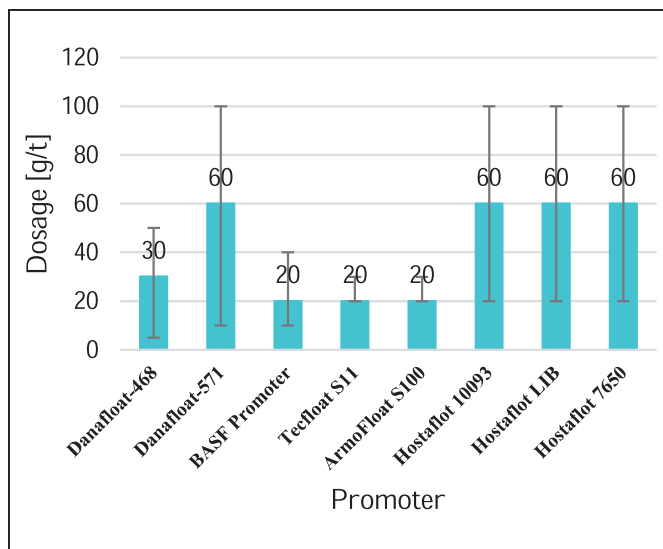


Figure 4. Dose levels of tested promoters at initial screening phase with range variations

Figure 4 illustrates the base dosage level (starting point) for each reagent and their dosage variation margins. These dosages were selected based on data sheets and producers' recommendations.

For the second phase of testing, a factorial experimental design focusing on 2 process variables (dosage of PAX and promoter) was established. An additional point of high promoter/low PAX dosage is added to evaluate whether PAX could be entirely excluded and substituted by a secondary promoter.

RESULTS AND DISCUSSION

Initial Screening of Reagents

The eight reagents have been compared against the base-case scenario (PAX-Aero-238) using a currently practiced flotation protocol. A specific objective is to find which combination will lead to a gold recovery higher than base case gold recovery. Figure 5 summarizes the average figures for gold recovery and concentrate mass yield obtained from the duplicate tests. The error bars indicate the highest and lowest points achieved with each reagent.

Review of the results undoubtedly ranks the Hostafloat promoters (10093, LIB, 7650) as the best performing ones—higher mass pull and recovery. The DP-OMC-1127 reached a recovery similar to the base-case scenario, however at a marginally lower dosage level of 20 g/t. Tecflore-S11 and Armofloate-S100 gave the worst performance (Au recovery of 81.2 and 81.7% respectively), a possible explanation being their low solubility, eventually requiring longer conditioning time. Therefore the four promoters selected for the second phase study were the three Hostafloat's and DP-OMC-1127. It should be noted, that the back-calculated feed assays for the tests were consistent.

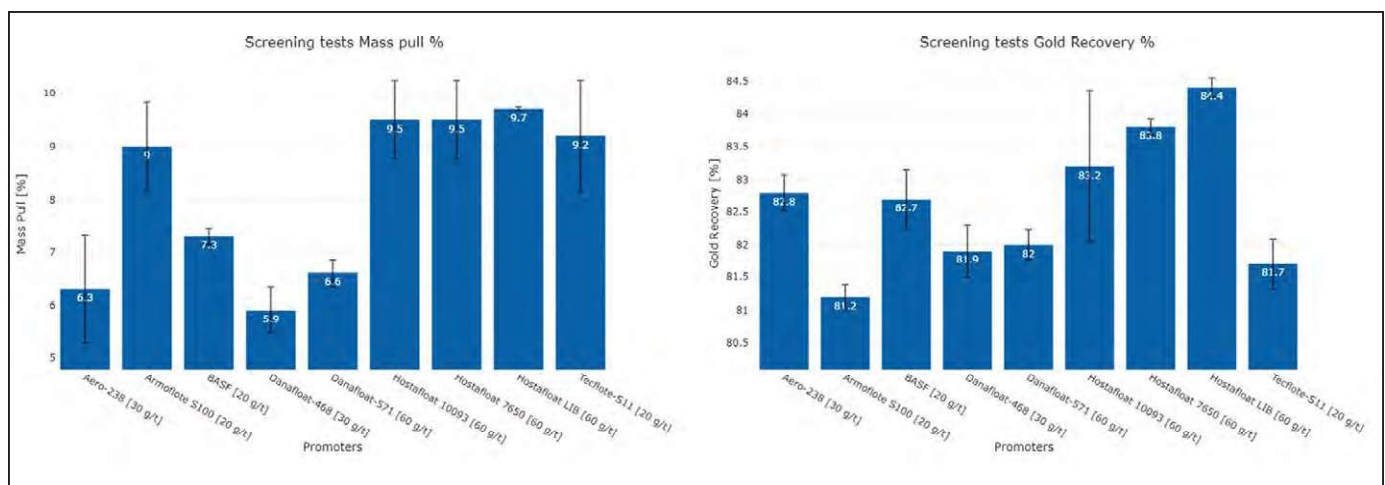


Figure 5. Gold recovery—comparative results from initial screening

The experimental design for the combination PAX-Promoter and their dose levels within the optimization trials is depicted in Figure 6. The PAX dose levels are always kept at three levels 280, 380 and 480 g/t. The scheme involves 4 points of high and low dosage collector and promoter, with the addition of a fifth point of high promoter/low collector dosage (low-right). For a sake of better visualization a sixth point (central) indicates the initial tests dose levels.

The central point dose levels for the three Hostafлот reagents is identical to the one for the initial tests. The low and high dosages for the promoters are selected based on their respective data sheets. The DP-OMC-1127 levels vary since their initial case dose is lower compared to the other promoters and the base-case (Aero-238). Moreover, the DP-OMC-1127 gave a recovery similar to that of Aero-238 during initial testing, therefore in view of eventual industrial application of this reagent, improved performance could be expected at higher dosage. No particular difference in the visual appearance of the froth has been noted during the tests with the various promoters.

Dose Levels Optimization

The results from the four reagents comparative performance are illustrated in Figure 7. The recovery of gold is visualized

in a 3D view depicting the 5 “optimization” tests from the factorial design and the test from the initial screening. Each point is an average from a duplicate realized with both batches of the ore.

For the DP-OMC-1127 performance, there is a clear optimal point at an inflexion point bringing Au recovery approaching 83.1% at 50–380 [Promoter-PAX] g/t dosage, a slightly higher figure compared with the base case of Aero-238 (82.8% Au recovery). On the other hand, the additional point at 50–380 [Promoter-PAX] g/t proves as not so efficient choice, delivering the second lowest recovery of 82.0%. For the Hostafлот-10093 the highest Au recovery of 86.4% is achieved at 100–380 [Promoter-PAX] g/t dosage, although the Au recovery increases concurrently with PAX and promoter dosage. For these experimental trials there is a clear trend of recovery dependence on the promoter dosage, also considering that the second highest recovery of 84.5% was obtained at 100–280 [Promoter-PAX] g/t. This results supports the possibility of replacing some of the PAX dosage with additional promoter supply.

Hostafлот-7650 reached 85.3% Au recovery at 100–280 [Promoter-PAX] g/t dosage, which was the highest recovery obtained. This is the only case from all testwork, where reaching a high recovery was possible by replacing a

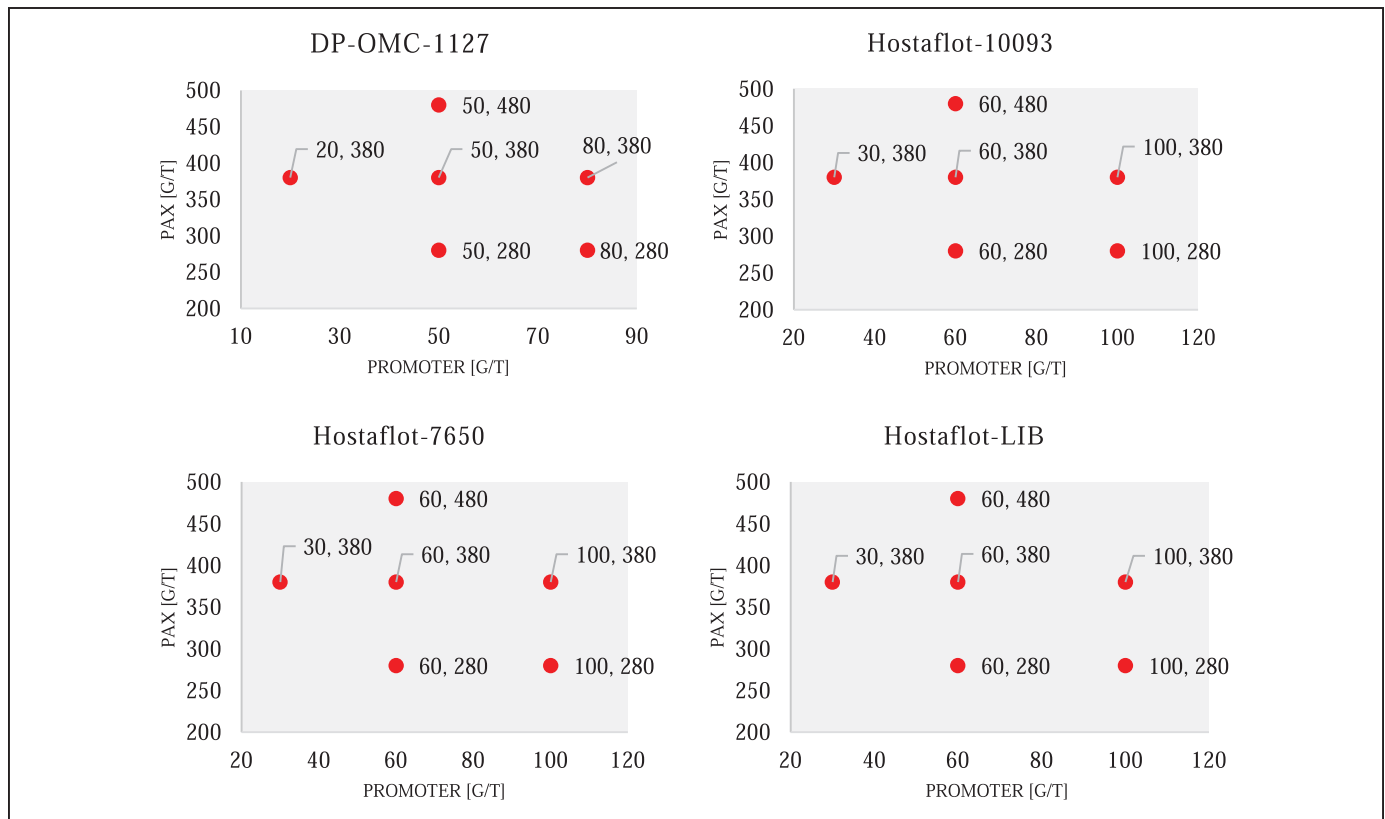


Figure 6. Experimental design for the four best performing promoters (variation in both collector and promoters dose levels)

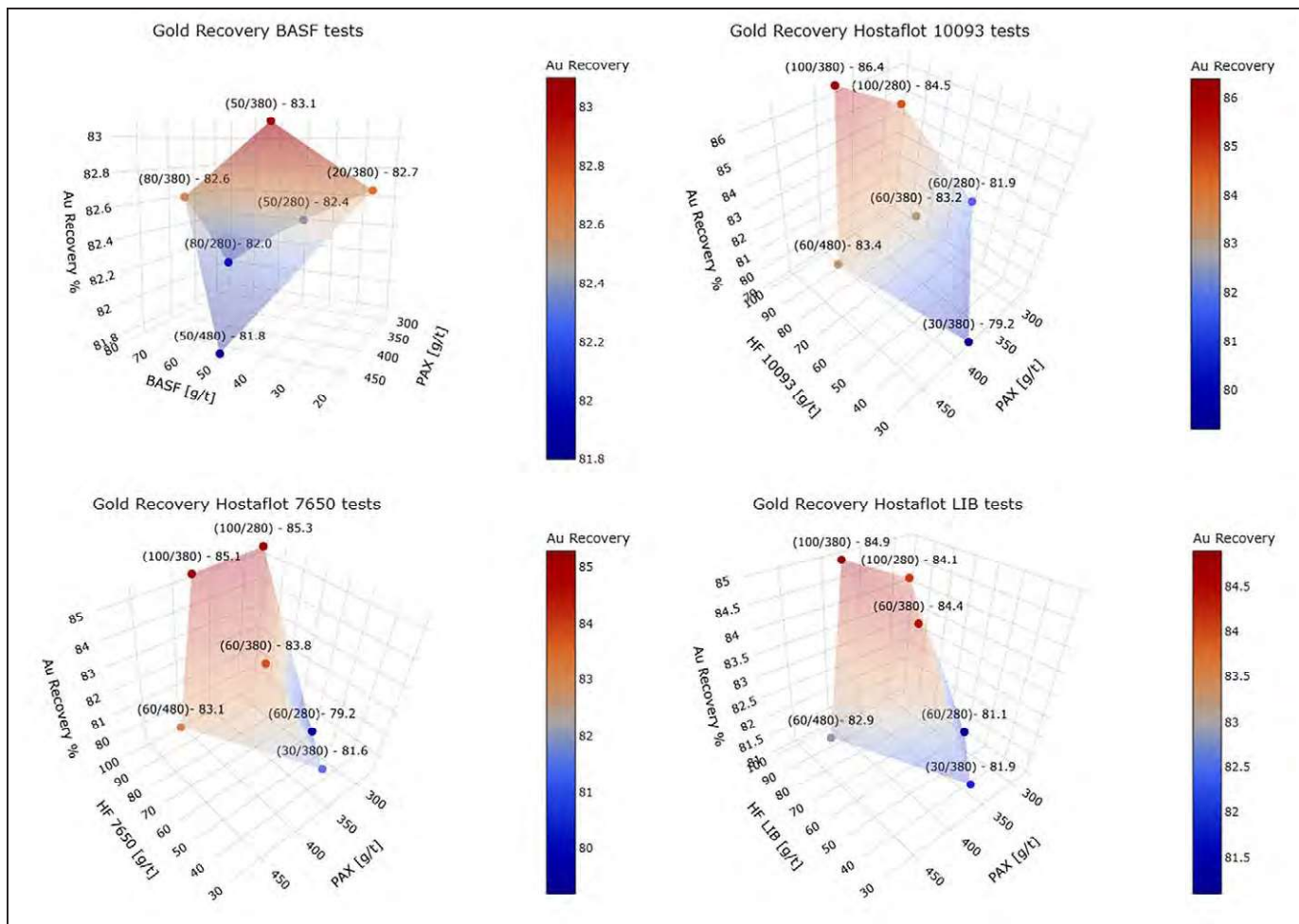


Figure 7. Gold recovery at varied collector and promoter dose levels for the four best performing reagents

fraction of the xanthate dosage with promoter. Hostafлот-LIB tests resulted in a maximum recovery of 84.9% at 100–380 [Promoter-PAX] g/t. The performance pattern here is similar to the one of Hostafлот-7650, although with slightly lower overall recovery. The main observation from the Hostafлот series is a linear behavior - promoter dose-Au recovery increase. Most likely an inflexion point is not yet reached, similar to the DP-OMC-1127 case, so better results at higher dosage could be achieved.

In order to better trace possible synergistic effect from the use of secondary collector, a correlative matrix has been constructed (Figure 8) to link Au, Ag and S recovery and mass pull as derived from the tests performed.

A correlation between mass pull and Au recovery could be established from the Hostafлот series results in contrast to those coming from the DP-OMC-1127 use. This means that for the Hostafлот reagents, the concentrate mass increase is leading to higher Au recovery. The overall mass pull is always higher for Hostafлот series tests (9.7–10.8%), than the one for the DP-OMC-1127 (8.1%). This observation

corroborates with the frothing features reported for the Hostafлот promoters, allowing the froth to stabilize and maintain higher mass pull in the concentrate fraction. This also means, that part of the concentrate material could have been recovered through entrainment rather than true flotation at higher dosages. The DP-OMC demonstrated slightly lower Au recovery, the improvement being entirely due to its increased dosage.

The above findings allowed for Hostafлот-7650 and Hostafлот-LIB to be ultimately recommended rather than Hostafлот-10093 and DP-OMC.

Au/Ag Ratio in Electrum and Effect on Flotation

One of the main objectives of the study was to investigate whether electrum grains richer in silver have a more pronounced tendency to report to the tailings. To this end, Figure 10 offers a comparative view of Au/Ag ratio as measured in the feed, concentrate and tailing products from selected tests.

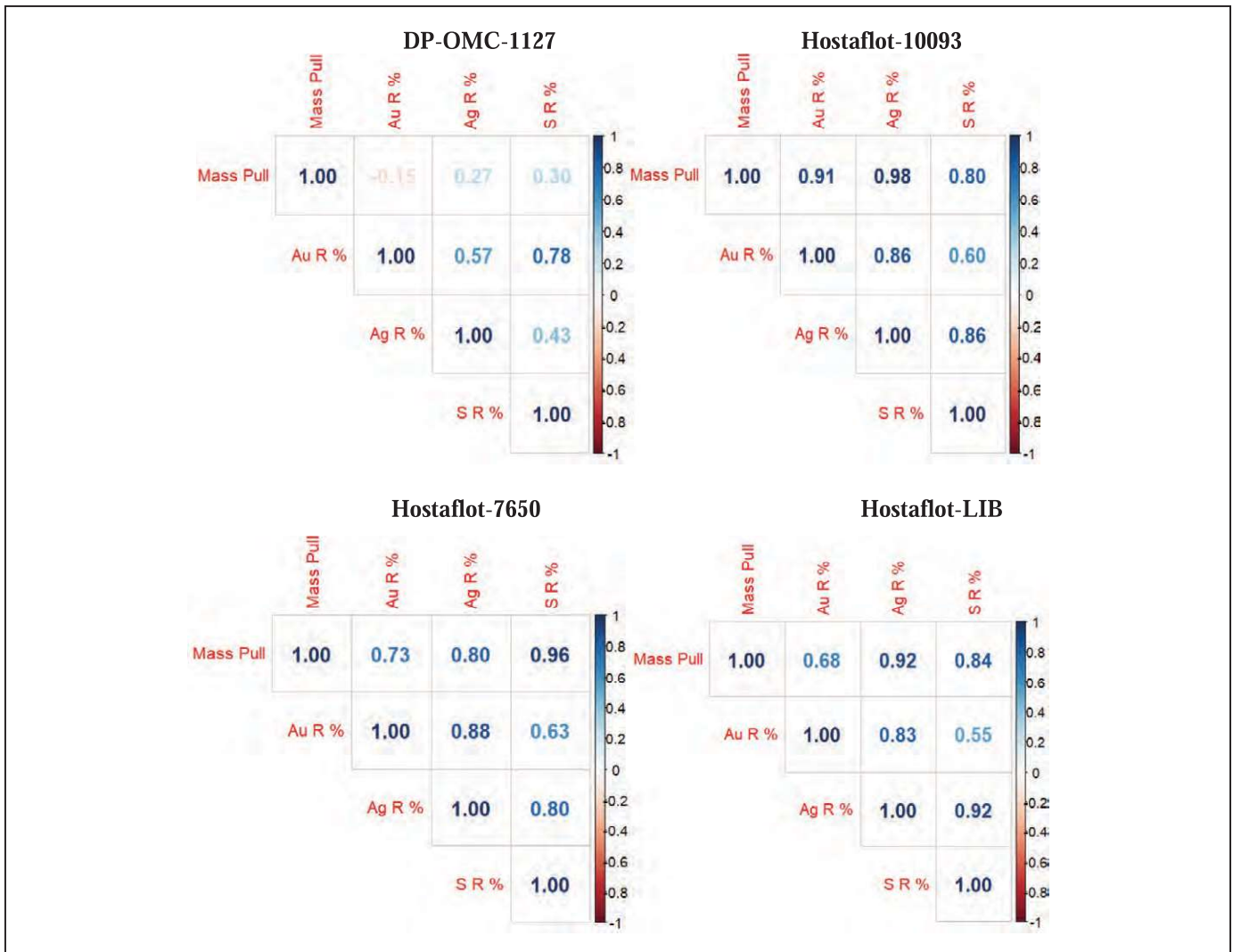


Figure 8. Correlation between Au, Ag and S recovery and mass pull (reagent optimization test trials)

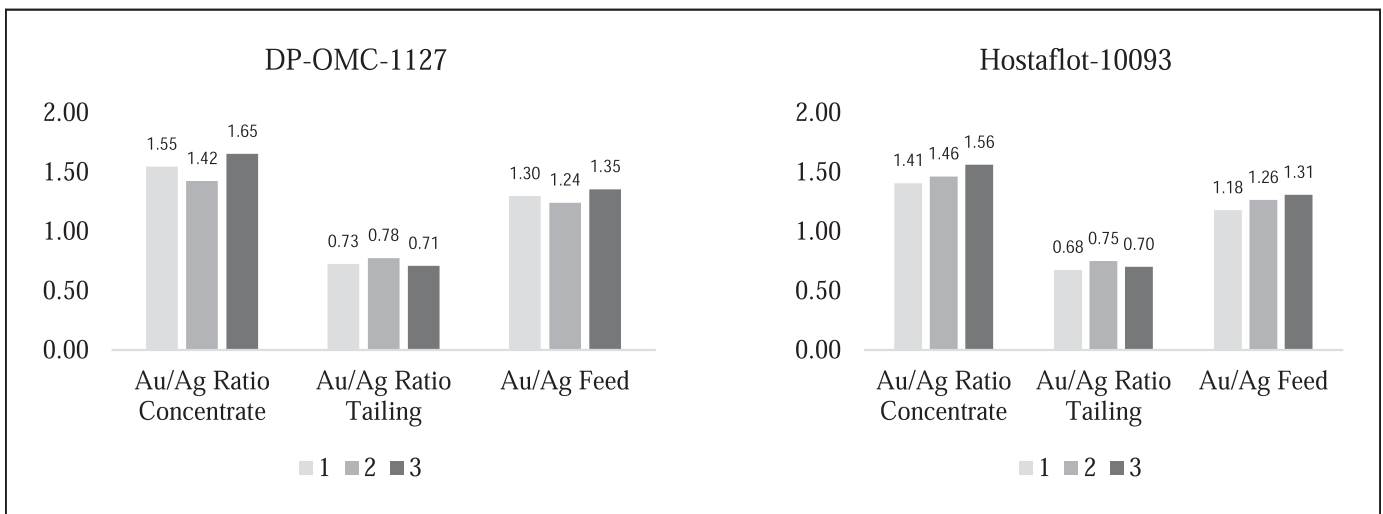


Figure 9. Comparison of Au/Ag ratio in feed, concentrate and tailings from reagent optimization series. DP-OMC- 50 g/t and Hostafлот-10093 - 60 g/t with PAX g/t, respectively at: 380 - 1, PAX 280 - 2, 480 - 3

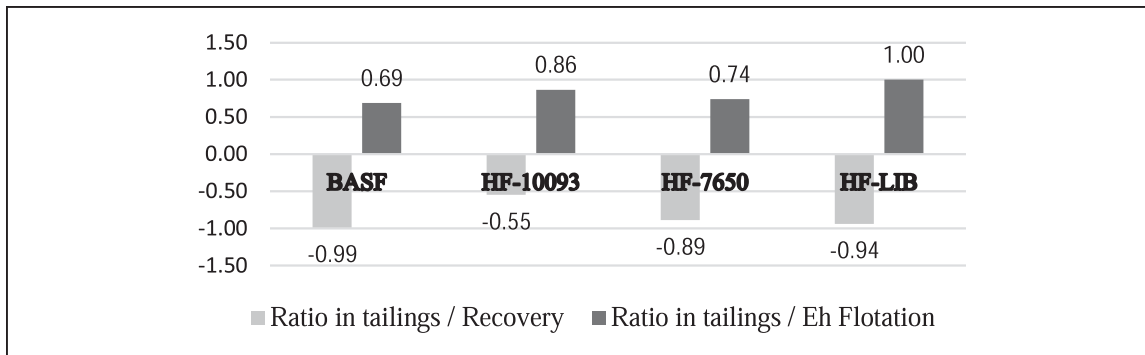


Figure 10. Correlation between Au/Ag ratio in electrum in tailings and Au recovery and Eh

One could observe that concentrates possess higher Au/Ag ratio than both feed and tailings. This implies that the tested reagents combinations deliver better gold recovery for the Au-rich electrum grains, with electrum found in tailings having Au grades below 44%.

An additional comparative analysis was done linking the Au/Ag ratio in tailings with the obtained recovery and the mean value of Eh registered during the tests. Figure 11 pictures the comparison between the 4 reagents subjected to dosage optimization, selecting 3 individual tests for each promoter, and correlating the results.

For the conclusions resulting from this comparison to be significant, several assumptions need to be taken into account. Firstly, gold is assumed to be present only in the form of electrum, meaning being associated with silver. Secondly, to enable a comparable correlation, the 3 tests selected are characterized by same dosage of promoter, with only the PAX dosage being varied. Thirdly, the Au/Ag ratio is different in each feed, for example the Au/Ag ratio in the feed of the DP-OMC tests varies between 1.24 to 1.35 and as such influences the ratio found in the concentrates and tailings. This situation could affect the correlation results. Therefore, the value of each Au/Ag ratio in tailings is normalized under their corresponding feed ratio, as follows.

$$\text{Normalized } \frac{\text{Au}}{\text{Ag}} \text{ ratio in tailings} = \frac{\frac{\text{Au}}{\text{Ag}}_{\text{Tailings}}}{\frac{\text{Au}}{\text{Ag}}_{\text{Feed}}}$$

Under the above described assumptions the correlation between Au/Ag ratio and recovery or Eh is possible. The results shown in Figure 11 suggest that as recovery of gold increases, so does the deportment of Au rich electrum in the concentrate and Ag rich electrum in the tailing. Additionally, according to results reported by Leppinen, 1991, the lower the potential, the higher the amount of silver rich electrum will be recovered. This however for a batch test poses some challenges explaining why the

remaining amount of electrum that remains in the tailing is predominantly Ag-rich.

Electrum grains occurrence and characteristics

It was important to find out the reasons for the occasional losses of precious metals in tailings and whether the type of particles could provide a clue. Therefore SEM-EDS inspection was performed on samples from the worst performing tests during reagent optimization, to observe size, morphology, liberation and Au/Ag ratio in the non-recovered grains. This approach could provide an explanation whether problematic metallurgical results are due to insufficient liberation or a low efficiency of reagents.

The SEM-EDS images shown in Figure 12 and 13 reveal electrum grains which are highly disseminated and have dimensions from 2 to 6 μm . Virtually all of them are attached to a much larger grain of quartz or calcite, reaching size up to 100 μm , as seen in Figure 12. The electrum found has a range of Au content from 74.7% to 84.8%, a proportion that is in a similar range to that found in the feed. Note that the tailings samples being imaged are from tests where gold recovery is below 80%. Logically the detected electrum grains in these tailings are often non-liberated and highly disseminated.

Given the characteristics of the detected electrum grains in the tailings (about 20 particles) and that no free grains were found, it is reasonable to assume that the liberated portion of electrum floats without hurdles under the tested reagent conditions. Hence to avoid occasional gold losses, the liberation of electrum trapped within the gangue need to be addressed. Given that a Vertimill is utilized at plant site to maintain P_{80} at 35 μm while the current lab-scale work employed a tumbling mill to reach similar ore granulometry, size and shape of the grains could be different under industrial conditions, hence a plant survey for mineralogical inspection need to be sought to affirm the current assumptions.

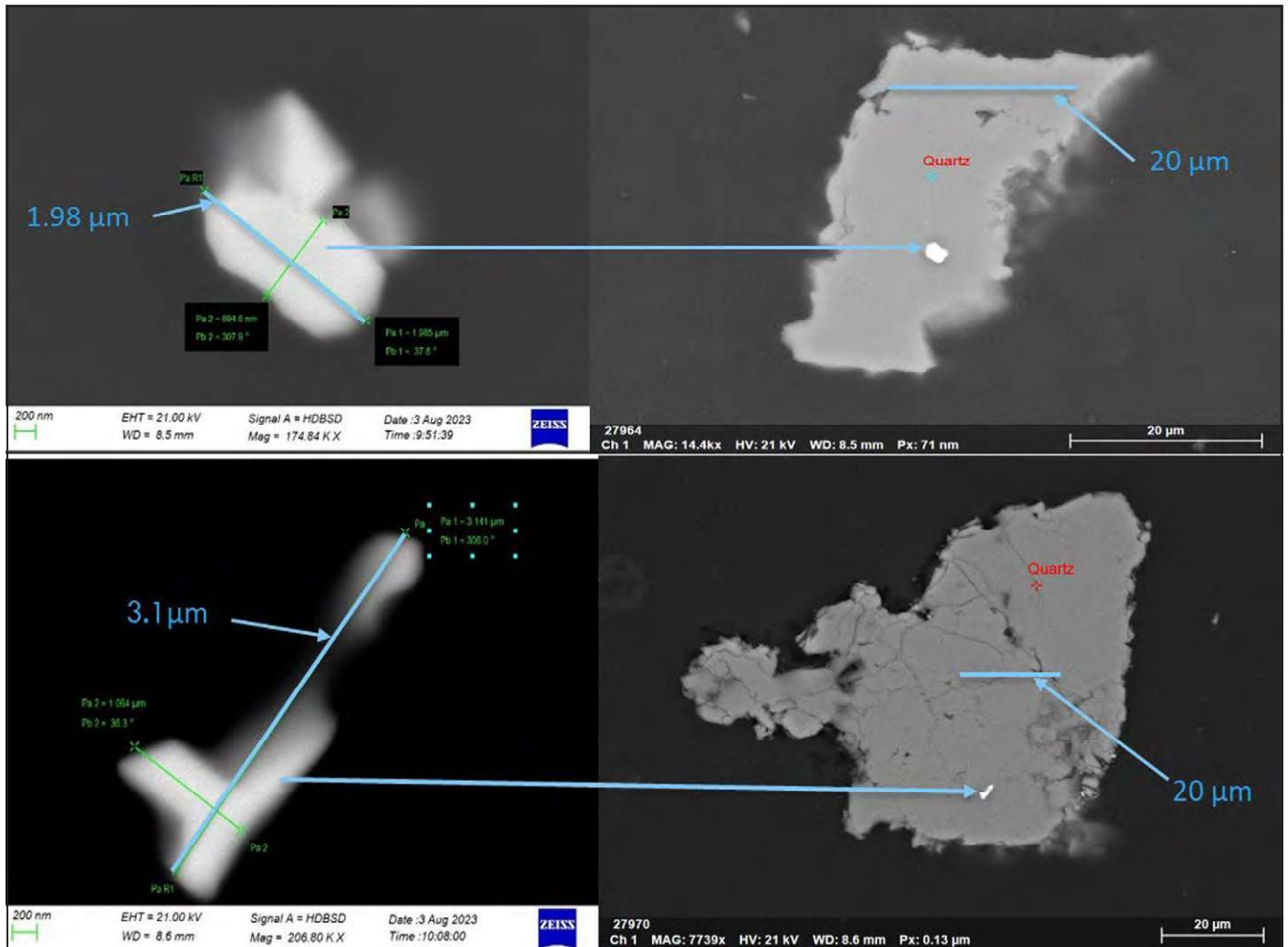


Figure 11. SEM backscattered electron images of grain met in tailings—test “Hostaflo 7650/PAX (60/280 g/t)”

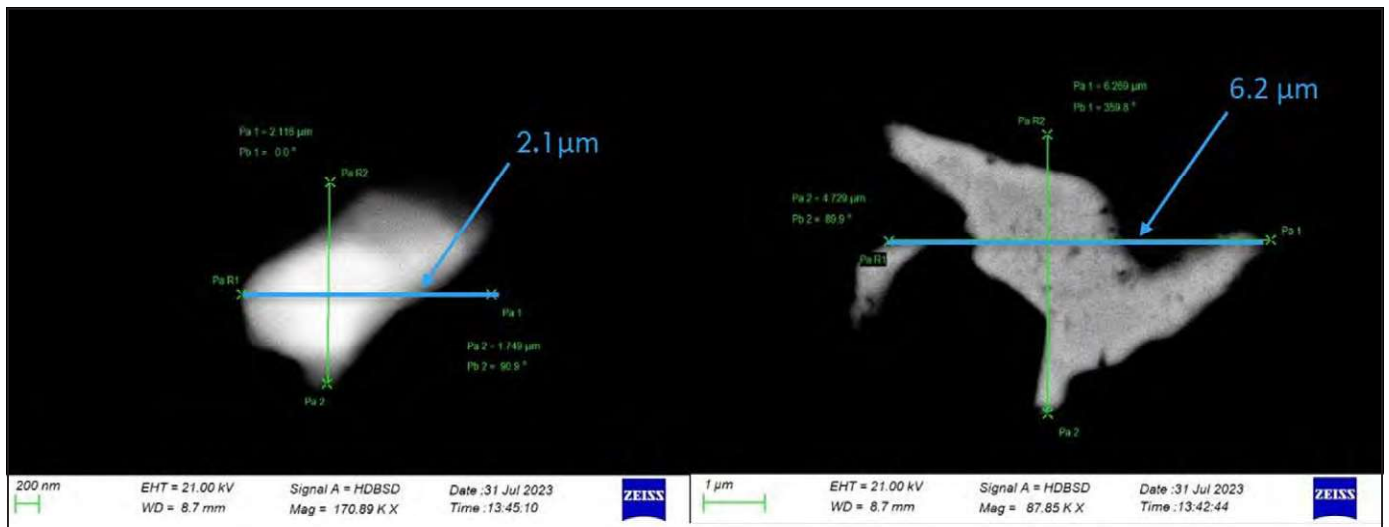


Figure 12. SEM backscattered electron images of grain met in tailings—test “Hostaflo LIB/PAX (60/280 g/t)”

CONCLUSIONS

Hostafлот-7650 with a dosage PAX/promoter of 280/100 g/t can be recommended against the currently used combination PAX/AERO238 at the Krumovgrad operation. The main supporting argument is that improved performance was obtained during a comparative testing campaign with this combination achieving 85.3% gold recovery, good reproducibility and with a reduced dosage of PAX.

The DP-OMC from BASF secured lower gold recovery than the Hostafлот series. However, the low correlation between mass pull and Au recovery suggests that most of the material is recovered thanks to true flotation and not mechanical entrainment.

Hostafлот-10093 gave the highest Au recovery from all tests (86.4%), but results are characterized by lower reproducibility as promoter dosage increases.

Hostafлот-LIB witnessed higher reproducibility compared to Hostafлот-7650 and Hostafлот-10093 and achieved comparably high recovery (84.9%). However at reduced PAX dosage, recovery dropped to 84.1%.

The Au/Ag ratio in electrum grains could be used as a metric for particles floatability, with the higher ratio of silver resulting in increased precious metals losses to tailings.

The SEM-EDS observations of grains detected in the streams suggest relatively good flotation response of the ore for the studied collector/promoter combinations. The reasons for Au losses in tailings are general the result of insufficient electrum liberation inside fine particle size classes.

The tailings samples are challenging when it comes to SEM-EDS detection of a statistically reliable number of electrum grains, therefore preconcentration through gravity separation is recommended for future analysis in order to increase the number of particles observed.

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